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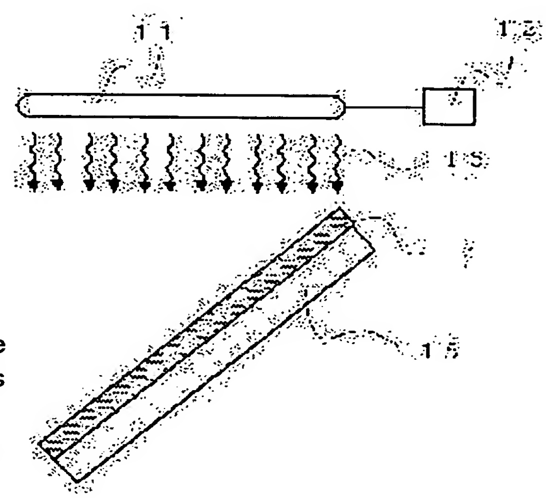
(54) RETARDATION FILM AND METHOD FOR MANUFACTURING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To realize a retardation film and a method for manufacturing the same by unpolarized ultraviolet rays irradiation.

SOLUTION: A mixture of a photosensitive polymer and a low molecular weight compound is applied (by spin coating or casting) to a substrate and film formed. Side chains in the photosensitive polymer and the low molecular weight compound are not oriented in the film, however, on irradiation of the unpolarized ultraviolet rays a photoreaction in a specified direction is suppressed, and at the same time with heating subsequent to the irradiation, orientation throughout the film takes place and birefringence is realized. Furthermore, as the inclination of the optical axis is freely set by the direction of the ultraviolet rays irradiation the film is useful as the retardation film enlarging a viewing angle of a liquid crystal display device.



LEGAL STATUS

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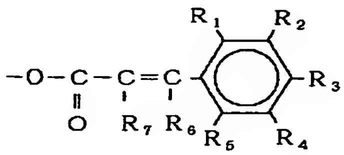
* NOTICES *

CLAIMS

[Claim(s)]
[Claim 1] a photosensitive polymer and the mixture of a low molecular weight compound -- the phase contrast film characterized by being produced at the process which includes the actuation which irradiates the light of unpolarized light nature in a film, and its manufacture approach.
[Claim 2] a photosensitive polymer and the mixture of a low molecular weight compound -- the phase contrast film characterized by being produced at the process which includes in a film the actuation which irradiates the light of unpolarized light nature from both front rear face, and its manufacture approach.
[Claim 3] the photosensitive polymer formed on the base material, and the mixture of a low molecular weight compound -- the phase contrast film characterized by being produced at the process which includes in a film the actuation which irradiates the light of unpolarized light nature from both directions on the back through a front face and a base material, and its manufacture approach.
[Claim 4] claim 1, claim 2, or claim 3 -- setting -- a photosensitive polymer and the mixture of a low molecular weight compound -- the phase contrast film characterized by being produced at the process in which the exposure of the light of the unpolarized light nature to a film includes the actuation irradiated from the direction of slant to a film plane, and its manufacture approach.
[Claim 5] The phase contrast film with which a photosensitive polymer has liquid crystallinity in claim 1, claim 2, claim 3, or claim 4, and its manufacture approach.
[Claim 6] The phase contrast film with which a low molecular weight compound has crystallinity or liquid crystallinity in claim 1, claim 2, claim 3, claim 4, or claim 5, and its manufacture approach.
[Claim 7] The phase contrast film on which a low molecular weight compound has bridge formation or the reactant radical which carries out a polymerization with light and/or heat in claim 1, claim 2, claim 3, claim 4, claim 5, or claim 6, and its manufacture approach.
[Claim 8] The phase contrast film characterized by being produced including the process heated and cooled in claim 1, claim 2, claim 3, claim 4, claim 5, a phase contrast film according to claim 6 or 7, and its manufacture approach, and its manufacture approach.
[Claim 9] The phase contrast film characterized by including the process which constructs a bridge in claim 1, claim 2, claim 3, claim 4, claim 5, claim 6, a phase contrast film according to claim 7 or 8, and its manufacture approach, and its manufacture approach.
[Claim 10] The phase contrast film characterized by having the structure both expressed with the photosensitive homopolymer or photosensitive copolymer whose principal chains which have the structure where a photosensitive polymer is expressed with a chemical formula 8 from a chemical formula 1 at least in claim 1 and claim 9, and which are expressed with a chemical formula 9 are a hydrocarbon, acrylate, methacrylate, maleimide, N-phenyl maleimide, a siloxane, etc., and its manufacture approach.
[Formula 1]



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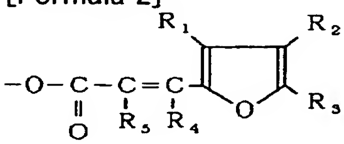
[Formula 2]



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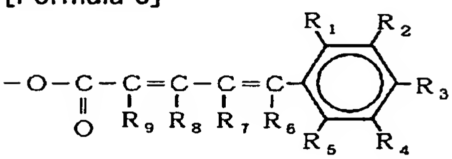
[Formula 3]



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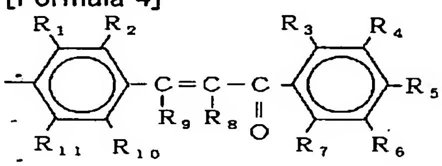

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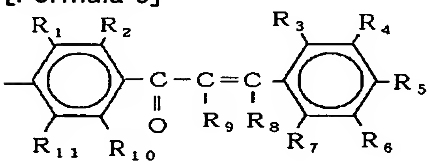

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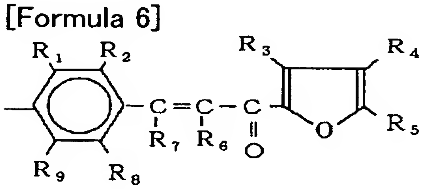

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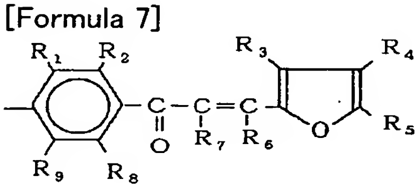
[Formula 7]



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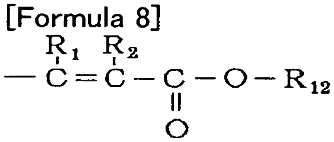
[Formula 8]



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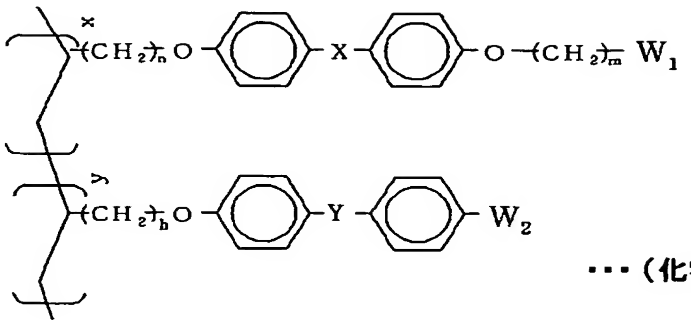
[Formula 9]



・・・（化学式9）

However, alkyloxy radicals, such as -R1 - -R11=-H, a halogen radical, -CN, an alkyl group, or a methoxy group, Or alkyl groups, such as a radical which carried out fluoridation of them, a -R12= methyl group, and an ethyl group, Y=none or the radical which carried out fluoridation of them -- it is -- x:y=100-0:0-100, n=1-12, m=1-12, and h= -- 1-12X -- - It is the structure expressed with COO, -OCO-, -N=N-, -C=C-or-C6H4-, W1, the W2= chemical formula 1, a chemical formula 2, a chemical formula 3, a chemical formula 4, a chemical formula 5, a chemical formula 6, a chemical formula 7, or a chemical formula 8.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] By irradiating the ultraviolet rays of unpolarized light nature at the film (film) of a photosensitive polymer and the mixture of a low molecular weight compound, it is related with the phase contrast film to which the optical axis was made to incline and orientation of the molecule was carried out, and its manufacture approach. (Especially the phase contrast film with which the optical axis inclined to the film plane is effective in angle-of-visibility expansion in a liquid crystal display.)
[0002]
[Description of the Prior Art] A phase contrast film is a film which has the birefringence which is made to penetrate the linearly polarized light component which vibrates in the perpendicular direction of a main shaft mutually, and gives phase contrast required among these 2 components. Such a phase contrast film is utilized also for the liquid crystal display field, and the phase contrast film with which especially the optical axis inclined is useful to angle-of-visibility expansion of a liquid crystal display as an optical compensation film. Some conventional techniques of manufacturing such a phase contrast film are reported. As one of them, polymeric materials, such as a

polycarbonate, are extended, orientation of the macromolecule chain is carried out, and although it is the refractive index of the extension direction, and a method of making the refractive index of the rectangular direction produce a difference to the extension direction, since a molecule carries out orientation in the extension direction, it is substantially impossible to make an optical axis incline. In view of the above-mentioned technical problem, an oriented film, rubbing, and the method of making a liquid crystallinity compound arrange on the base material which carried out orientation processing by optical exposure are being proposed or put in practical use as a manufacturing method of the phase contrast film with which the optical axis inclined. For example, the method of making discotheque liquid crystal arrange on the rubbing orientation film and the method vacuum evaporationo orientation film of SiO slanting is indicated by JP,7-287119,A and JP,7-287120,A. Moreover, the approach of making carry out orientation of the discotheque liquid crystal containing a photopolymerization initiator, and fixing this orientation by optical exposure on the optical orientation film, is indicated by JP,10-278123,A as same approach. By the approach using the above orientation film, there are problems, like processes, such as orientation processing of the orientation film and orientation of a liquid crystal ingredient, become complicated. Furthermore, although the approach of carrying out the method vacuum evaporationo of the inorganic dielectric of slanting is proposed as other methods of manufacturing the phase contrast film with which the optical axis inclined, in order to form the vacuum evaporationo film succeeding a long picture-like sheet top, equipment becomes large-scale or there are problems, like a process becomes complicated. Although the approach of irradiating [JP,7-138308,A] (UV) light of linearly polarized light nature at photosensitive polymers, such as polyvinyl cinnamate, is indicated as an approach of making phase contrast discovering by optical exposure, since an anisotropy is discovered to electric-field vibration and perpendicularly of polarization UV light it irradiated and an optical axis cannot be leaned, by this approach, it is hard to expand an angle of visibility. Moreover, this invention person also proposed the approach of manufacturing the phase contrast film with which the optical axis inclined by the UV irradiation of the linearly polarized light nature to the side-chain mold liquid crystallinity macromolecule which has photosensitivity, in JP,10-278123,A. However, it is necessary to change the ultraviolet rays of unpolarized light nature into linearly polarized light nature, and to irradiate them by the approach of making phase contrast discovering, by optical exposure. There are some which sandwiched what infiltrated iodine into the sheet which carried out uniaxial stretching of the PVA (polyvinyl alcohol) as a common dichroic polarizer used for such polarization conversion by TAC (triacetyl cellulose). However, in the dichroic polarizer into which iodine was infiltrated in this way, since the permeability and thermal resistance of the light of an ultraviolet region are low, use is not borne as a liquid crystal light orientation technique. Since it is such, birefringence mold prism is used to polarize the light of an ultraviolet region, but in order to use the natural crystal of a calcite as prism by birefringence mold prism, there is no large-sized prism which can irradiate the whole substrate surface which is used for LCD.

[0003]

[Problem(s) to be Solved by the Invention] In order that a molecule may carry out orientation in the extension direction with the phase contrast film produced by the extension orientation of a high polymer film, it is remarkably difficult to make an optical axis incline. Although it is possible to produce the phase contrast film which made the optical axis incline, since a process becomes complicated, neither the approach of on the other hand making a liquid crystallinity compound arranging on the base material which carried out orientation processing, nor the approach of carrying out the method vacuum evaporationo of the inorganic dielectric of slanting is easy for obtaining the phase contrast film which made the optical axis of a large area incline by low cost. Moreover, although it is necessary to make exposure light into the linearly polarized light through a polarizing element by the approach of manufacturing a phase contrast film by the UV irradiation of linearly polarized light nature, it is difficult to produce the practical polarizing element in the case of irradiating a large area.

[0004]
[Means for Solving the Problem] In this invention, it is related with the phase contrast film which made the optical axis incline and made phase contrast discover by irradiating the ultraviolet rays of unpolarized light nature at the film (film) of a photosensitive polymer and the mixture of a low molecular weight compound. By the manufacture approach (phase contrast film to twist) of the phase contrast film of this invention, by producing a photosensitive polymer and the mixture of a low molecular weight compound, and irradiating the ultraviolet rays of unpolarized light nature with a specific tilt angle to this film surface, lean in the tilt-angle direction of the ultraviolet rays of this unpolarized light nature that irradiated the molecule in the film, it is made to carry out orientation, and the phase contrast film which set up the optical axis towards desired can be offered.
[0005]
[Embodiment of the Invention] Below, the detail of this invention is explained. Substituents, such as a biphenyl currently used abundantly as a meso gene component of a liquid crystallinity giant molecule as an example of the above-mentioned photosensitive polymer, terphenyl, phenyl benzoate, and an azobenzene, A cinnamoyl radical, a chalcone radical, the Singh Nami Liden radical, beta-(2-furil) acryloyl radical () Or the macromolecule which has a side chain including the structure which combined photosensitive radicals, such as those derivatives, and has structures, such as a hydrocarbon, acrylate, methacrylate, maleimide, N-phenyl maleimide, and a siloxane, in a principal chain is mentioned. It is also possible to carry out copolymerization of the unit which has the side chain which the copolymer of the unit which has the side chain with which the homopolymer which consists of the same repeat unit differs from structure is sufficient as this polymer, or does not contain a photosensitive radical. Moreover, the compound which has the crystallinity which has substituents, such as a biphenyl by which the low molecular weight compound to mix is also used abundantly as a meso gene component, terphenyl, phenyl benzoate, and an azobenzene, or liquid crystallinity is mentioned. It does not restrict but the low molecular weight compound to mix can also mix two or more sorts of compounds with a single compound. Furthermore, adding the cross linking agent for raising the orientation assistant for raising a stacking tendency to extent which does not spoil liquid crystallinity, and thermal resistance, and the monomer which does not show liquid crystallinity, without spoiling liquid crystallinity may be copolymerized in a photosensitive polymer. However, a photosensitive polymer and a photosensitive low molecular weight compound are not limited above.
[0006] An example is given and this invention is explained in detail. A photosensitive polymer and the mixture of a low molecular weight compound are applied (a spin coat thru/or cast), and a film is produced. This film is isotropy at the time of film production, and the photosensitive side-chain section and the photosensitive low molecular weight compound of a polymer have not turned to the specific direction. This condition is explained based on drawing 2 . Drawing 2 expresses the film after film production typically. In the film (20), the low molecular weight compound (2c) shown with the side chain (2a, 2b) and cylinder which have the sensitization radical shown with prolate ellipsoid exists disorderly (coexistence). If the ultraviolet-rays false parallel flux of light (L) of unpolarized light nature is irradiated at this film, since the side chain (2a) suitable for the travelling direction and perpendicular direction of exposure ultraviolet rays tends to be exposed in parallel from the suitable side chain (2b), the photoreaction will progress alternatively. This is because the conjugated system of the photosensitive part containing the benzene ring etc. is prolonged in the direction of a major axis of a side chain, and when such a side chain is put on emanating space like light, it is because an interaction serves as the maximum when the electric-field oscillating direction of light is in agreement with the direction of a major axis of a side chain, and an interaction serves as the minimum when the travelling direction of light and the direction of a major axis of a side chain are in agreement. From this, it can consider as the film which controlled the photoreaction of the specific direction by the exposure of the ultraviolet rays of unpolarized light nature. In order to advance this photoreaction, the exposure of the light of wavelength to which the part of a photosensitive radical

can react is required. Although this wavelength changes also with classes of photosensitive radical, generally it is 200 to 500 nm, and its effectiveness of 250 to 400 nm is high especially in many cases.

[0007] Drawing 3 shows the mimetic diagram of the film with which the orientation after irradiating the ultraviolet rays of unpolarized light at the film of drawing 2 was promoted. As shown in drawing 3 , the side chain (3d) or low molecular weight compound (3c) which did not start the photoreaction in the film (30) by the molecular motion after the exposure of the ultraviolet rays of unpolarized light nature, orientation of the effect of the anisotropy discovered in the film is influenced and carried out by having controlled the photoreaction of the specific direction (the side chain which exposed the side chain (3a) by unpolarized light exposure, and reacted --) the side chain (3b) expresses the side chain which did not react since the travelling direction and parallel direction of exposure ultraviolet rays were turned to, respectively. . Consequently, in the whole film, it can lean to the travelling direction of the ultraviolet rays of unpolarized light nature, it can be made to carry out orientation, and induction of the birefringence is carried out. If this exposure is performed from across to a film surface, since arbitration can be made to be able to incline and orientation of the optical axis can be carried out, the phase contrast film which set up the optical axis towards desired can be offered. measurement of the inclination of an optical axis -- Japan Journal Applied Physics and Vol. -- the crystal rotation method which measures the transparency reinforcement of polarization was used, rotating the test portion carried by 19 and 2013 (1980). By this measuring method, measurement of the three-dimensional birefringence of a test portion can be performed from the angular dependence of the permeability of polarization. Heating of the film promotes the orientation by the molecular motion after the exposure of the ultraviolet rays of unpolarized light nature. Whenever [membranous stoving temperature] is lower than the softening temperature of the part which carried out the photoreaction, and it is desirable that it is higher than the side chain which did not carry out the photoreaction, and low-molecular softening temperature. Moreover, it is also effective to irradiate the ultraviolet rays of unpolarized light nature under heating, for promoting membranous orientation (from a room temperature to Ti+5 degree C). Here, Ti points out the phase transition temperature when changing from a liquid crystal phase to an isotropic phase. It is effective to irradiate the ultraviolet rays of unpolarized light nature before and behind Ti preferably. Thus, if the film which irradiated the ultraviolet rays of unpolarized light nature under heating or heating is cooled below to the softening temperature of this macromolecule after irradiating the ultraviolet rays of unpolarized light nature, a molecule will be frozen and the phase contrast film of this invention will be obtained.

[0008] When the low molecular weight compound mixed to a photosensitive polymer in this invention has heat and/or photoreaction nature to low molecular weight compounds or this polymer, since orientation is fixed firmly, heat-resistant improvement is expected. In such a case, it is necessary to stop light exposure, or to adjust reactivity and to control the consistency of a photoreaction point not to bar the molecular motion at the time of reorientation. While a low molecular weight compound has the effectiveness which will bloom cloudy if it is optimum dose, and controls whenever, if it adds superfluously, it will bloom cloudy, and causes the increment in whenever, and the fall of a stacking tendency. although based also on a photosensitive polymer or the class of low molecular weight compound from such a viewpoint -- a low molecular weight compound -- 0.1wt(s)% - 80wt% -- although a phase contrast film can be manufactured even if it adds, it is desirable that it is 5wt(s)% - 50wt% preferably. Here, when the compatibility of a polymer and a low molecular weight compound is not enough, and a film is produced, with heating of the film after irradiating the ultraviolet rays of unpolarized light nature, the crystal of the magnitude which can carry out induction of the dispersion of phase separation or the light is generated, and it becomes the cause of the increment in whenever [cloudy].

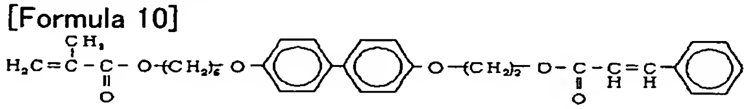
[0009] As the technique of thickening thickness and obtaining the film of bigger phase contrast, the approach of carrying out the laminating of the film is mentioned. In this case, it is the process which

applies and carries out the laminating of the ingredient solution on the film which produced the film previously and irradiated the ultraviolet rays of unpolarized light nature, and in order to prevent destruction of this film formed previously, it is effective to dissolve and use a polymer and a low molecular weight compound for the solvent which lowered solubility. Moreover, a birefringence will come to be more efficiently discovered if the ultraviolet rays of unpolarized light nature are irradiated from a front rear face at the film of a photosensitive compound. A photosensitive compound is applied on a base material, and is produced, and the exposure of the ultraviolet rays of unpolarized light nature may mind direct or a base material. In minding a base material, although what kind of ingredient is sufficient as a base material as long as it has the permeability of the light of wavelength to which a photosensitive compound can react, there are so few exposures that light transmittance is high, it ends, and becomes advantageous on a production process. Moreover, a compound photosensitive in the base material top of detachability can be produced, and the ultraviolet rays of unpolarized light nature can also be irradiated from a membranous front rear face after exfoliation.

[0010] The synthetic approach about the raw material compound and low molecular weight compound of a photosensitive polymer is shown below.

(Monomer 1) The 4-hydroxy-4'-hydroxy ethoxy biphenyl was compounded by heating 4, and 4'-biphenyl diol and 2-chloroethanol under alkali conditions. 1 and 6-dibromo hexane was made to react to this product under alkali conditions, and the 4-(6-BUROMO hexyloxy)-4'-hydroxy ethoxy biphenyl was compounded. Subsequently, lithium methacrylate was made to react and the 4-hydroxy ethoxy-4'-(6-methacryloyl hexyloxy) biphenyl was compounded. Finally, the phenylacrylyl chloride was added to the bottom of a basic condition, and the monomer 1 shown in a chemical formula 10 was compounded.

[Formula 10]



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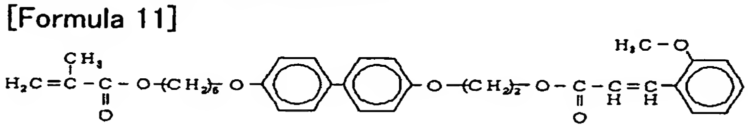
[0011] (Monomer 2) The 4-hydroxy-4'-hydroxy ethoxy biphenyl was compounded by heating 4, and 4'-biphenyl diol and 2-chloroethanol under alkali conditions. 1 and 6-dibromo hexane was made to react to this product under alkali conditions, and the 4-(6-BUROMO hexyloxy)-4'-hydroxy ethoxy biphenyl was compounded. Subsequently, lithium methacrylate was made to react and the 4-hydroxy ethoxy-4'-(6-methacryloyl hexyloxy) biphenyl was compounded. Finally, 2-methoxy phenylacrylyl chloride was added to the bottom of a basic condition, and the monomer 2 shown in a chemical formula 11 was compounded.

[Formula 11]



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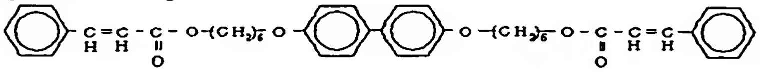
[0012] (Polymer 1) This monomer 1 was dissolved into the tetrahydrofuran and the photosensitive polymer 1 was obtained by adding and carrying out the polymerization of the azobisuisobutironitoriru (azobisisobutyronitril) as a reaction initiator. This polymer 1 presented liquid crystallinity in the temperature field of 47 to 75 degree C.

[0013] (Polymer 2) This monomer 2 was dissolved into the tetrahydrofuran and the photosensitive polymer 2 was obtained by adding and carrying out the polymerization of the azobisuisobutironitoriru as a reaction initiator. This polymer 2 also presented liquid crystallinity.

[0014] (Low molecular weight compound 1) 4, and 4'-biphenyl diol and 6-BUROMO hexanol are made to react under alkali conditions, and they are 4 and 4'. - The screw (6-BUROMO hexyloxy)

biphenyl was compounded. Subsequently, add a phenylacrylyl chloride, it was made to react to the bottom of a basic condition, and the low molecular weight compound 1 shown in a chemical formula 12 was compounded by carrying out column purification of the product.

[Formula 12]



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[0015] An example is given in drawing 1 and the manufacture approach (equipment) of the phase contrast film of this invention is shown in it. The film (14) of the photosensitive polymer to which the ultraviolet rays (16) of the unpolarized light nature generated with the ultraviolet ray lamp (11) excited by the power source (12) were applied on the base material (15) (coat), and the mixture of a low molecular weight compound is irradiated. Examples 1-3 are examples which produced the phase contrast film to which the optical axis inclined according to the manufacturing method of this invention.

[0016] (Example 1) 3.75% of the weight of the polymer 1 and 1.25% of the weight of the liquid crystal ingredient E7 (Merck Japan) were dissolved in the dichloroethane, and it applied by the thickness of about 3 micrometers on the quartz substrate (base material). This substrate was leaned 45 degrees to the horizontal plane, it has arranged so that a spreading side may turn into an exposure side, and the ultraviolet rays of unpolarized light nature were perpendicularly irradiated two times 120 mJ/cm at the room temperature to the horizontal plane, then the ultraviolet rays of unpolarized light nature were irradiated to the substrate two times 120 mJ/cm inside out similarly. Next, after heating at 100 degrees C, it cooled to the room temperature. Thus, as for the obtained substrate, 20 degrees of opticals axis inclined from [of a substrate] the normal, and the phase contrast within a substrate side was 14nm.

[0017] (Example 2) 3.75% of the weight of the polymer 2 and 1.25% of the weight of the liquid crystal ingredient E7 (Merck Japan) were dissolved in the dichloroethane, and it applied by the thickness of about 3 micrometers on the quartz substrate (base material). This substrate was leaned 45 degrees to the horizontal plane, it has arranged so that a spreading side may turn into an exposure side, and the ultraviolet rays of unpolarized light nature were perpendicularly irradiated two times 120 mJ/cm at the room temperature to the horizontal plane, then the ultraviolet rays of unpolarized light nature were irradiated to the substrate two times 120 mJ/cm inside out similarly. Next, after heating at 100 degrees C, it cooled to the room temperature. Thus, as for the obtained substrate, 25 degrees of opticals axis inclined from [of a substrate] the normal, and the phase contrast within a substrate side was 20nm.

[0018] (Example 3) 3.75% of the weight of the polymer 1 and 1.25% of the weight of the low molecular weight compound 1 were dissolved in the dichloroethane, and it applied by the thickness of about 3 micrometers on the quartz substrate (base material). This substrate was leaned 45 degrees to the horizontal plane, it has arranged so that a spreading side may turn into an exposure side, and the ultraviolet rays of unpolarized light nature were perpendicularly irradiated two times 120 mJ/cm at the room temperature to the horizontal plane, then the ultraviolet rays of unpolarized light nature were irradiated to the substrate two times 120 mJ/cm inside out similarly. Next, after heating at 100 degrees C, it cooled to the room temperature. Thus, as for the obtained substrate, 7 degrees of opticals axis inclined from [of a substrate] the normal, and the phase contrast within a substrate side was 1nm.

[0019] By irradiating ultraviolet rays further, the phase contrast film with which the optical axis produced by these examples inclined can be made to be able to promote the photoreaction of an unreacted photosensitive radical, and the orientation in a film can be firmly fixed to it. Such a phase contrast film was excellent in thermal resistance and light stability, and enough for practical use. Even if it could produce the film which controlled the optical axis by irradiating the ultraviolet rays

of unpolarized light nature and did not use a practical polarizing element like the conventional technique from these examples, it was proved that manufacture of the phase contrast film which controlled the direction of an optical axis effective in angle-of-visibility expansion of a liquid crystal display is possible.

[0020]

[Effect of the Invention] By simple actuation of the exposure of the ultraviolet rays of unpolarized light nature, even if it does not use an extension process like the conventional technique, a phase contrast film can be obtained. Furthermore, by changing the direction of radiation of the ultraviolet rays of unpolarized light nature, production of the field where opticals axis differ in the same substrate is also possible, and the activity to various optical elements is expected. Moreover, it can twist and the phase contrast film with which the optical axis inclined can be utilized as an optical compensation film for angle-of-visibility expansion in the liquid crystal display using rotatory-polarization mode and birefringence mode using a nematic liquid crystal. Although such [conventionally] a phase contrast film of a large area with which the optical axis inclined was unproducible by low cost, large area-ization was attained from across by simple actuation of irradiating the ultraviolet rays of unpolarized light nature, by this invention.

[0021]

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The conceptual diagram showing the manufacture approach of the phase contrast film of this invention

[Drawing 2] The mimetic diagram showing the side chain exposed by the exposure of the ultraviolet rays of unpolarized light nature

[Drawing 3] The mimetic diagram showing the film which carried out orientation by the molecular motion after irradiating the ultraviolet rays of unpolarized light nature

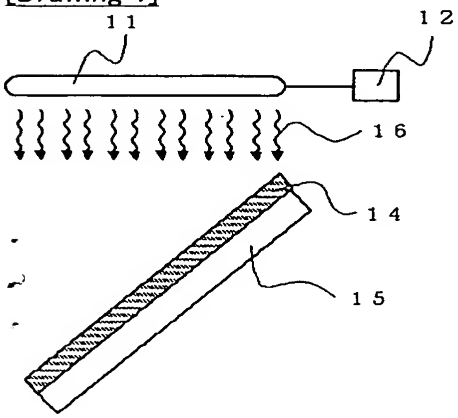
[Description of Notations]

11 ... Ultraviolet ray lamp
12 ... Power source
14 ... Film (film)
15 ... Base material (base material)
16 ... Ultraviolet rays (light of unpolarized light nature)

[Translation done.]

DRAWINGS

[Drawing 1]



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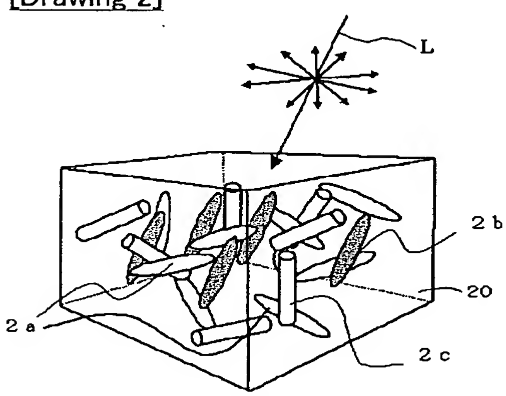

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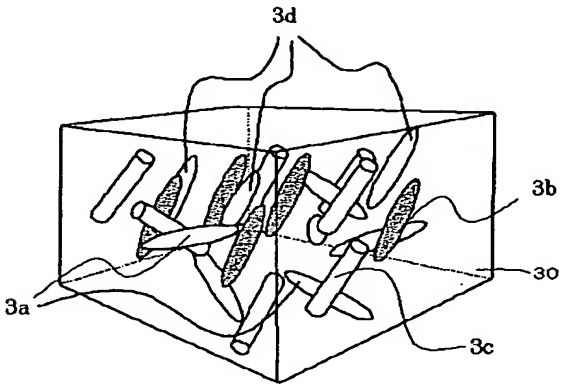

[Translation done.]

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                  CEZ
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   G03F  7/004    521
         7/038    501
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【FI】

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   G02B  5/30
   C08F  2/48
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   G03F  7/004    521
         7/038    501
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【審査請求】未請求
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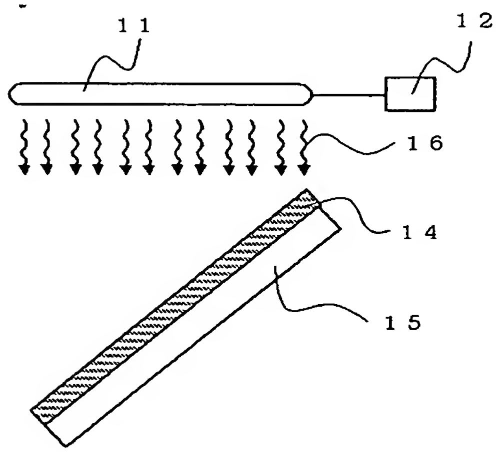
要約

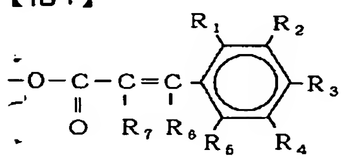
(57)【要約】
【目的】非偏光性の紫外線の照射による位相差フィルム、およびその製造方法の実現。
【構成】感光性の重合体と低分子化合物の混合体を塗布(スピンコートないしキャスト)しフィルム化する。このフィルム中で感光性の重合体の側鎖と低分子化合物は配向していないが、非偏光性の紫外線を照射すると、特定方向の光反応が抑制されると共に、照射後の加熱によってフィルム全体に亘って配向し複屈折を発現する。更に、紫外線の照射方向により光軸の傾きを自由に設定できるので、液晶表示装置の視野角を拡大する位相差フィルムとして役立つ。



請求の範囲

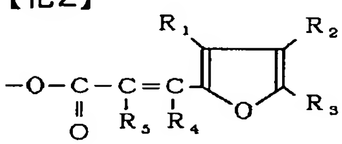
【特許請求の範囲】
【請求項1】感光性の重合体と低分子化合物の混合体フィルムに非偏光性の光を照射する操作を含む工程で作製されることを特徴とする、位相差フィルムおよびその製造方法。
【請求項2】感光性の重合体と低分子化合物の混合体フィルムに表裏面両方から非偏光性の光を照射する操作を含む工程で作製されることを特徴とする、位相差フィルムおよびその製造方法。
【請求項3】支持体上に形成された感光性の重合体と低分子化合物の混合体フィルムに表面および支持体を介して裏面の両方向から非偏光性の光を照射する操作を含む工程で作製されることを特徴とする、位相差フィルムおよびその製造方法。
【請求項4】請求項1、請求項2または請求項3において、感光性の重合体と低分子化合物の混合体フィルムへの非偏光性の光の照射がフィルム面に対し斜め方向より照射する操作を含む工程で作製

されることを特徴とする、位相差フィルムおよびその製造方法。
【請求項5】請求項1、請求項2、請求項3または請求項4において、感光性の重合体が液晶性を有する位相差フィルムおよびその製造方法。
【請求項6】請求項1、請求項2、請求項3、請求項4または請求項5において、低分子化合物が結晶性または液晶性を有する位相差フィルムおよびその製造方法。
【請求項7】請求項1、請求項2、請求項3、請求項4、請求項5または請求項6において、低分子化合物が光および／または熱により架橋または重合する反応性基を有する位相差フィルムおよびその製造方法。
【請求項8】請求項1、請求項2、請求項3、請求項4、請求項5、請求項6または請求項7に記載の位相差フィルムおよびその製造方法において、加熱ならびに冷却する工程を含んで、作製されることを特徴とする位相差フィルムおよびその製造方法。
【請求項9】請求項1、請求項2、請求項3、請求項4、請求項5、請求項6、請求項7または請求項8に記載の位相差フィルムおよびその製造方法において、架橋する工程を含むことを特徴とする位相差フィルムおよびその製造方法。
【請求項10】請求項1および請求項9において感光性の重合体が少なくとも化学式1から化学式8で表される構造を有する共に、化学式9で表される主鎖が炭化水素、アクリレート、メタクリレート、マレイミド、Nーフェニルマレイミド、シロキサンなどである感光性の単独重合体または共重合体で表される構造を有することを特徴とする位相差フィルムおよびその製造方法。
【化1】



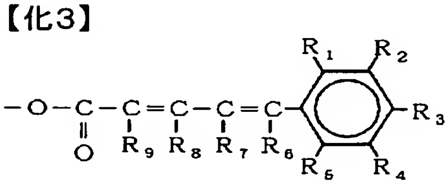
･･･（化学式1）

【化2】



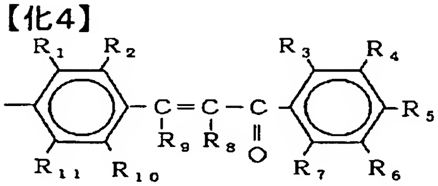
･･･（化学式2）

【化3】



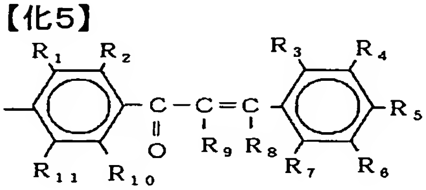
･･･（化学式3）

【化4】



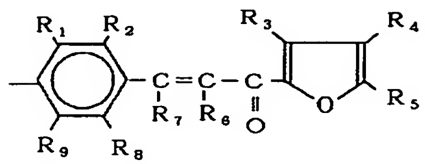
･･･（化学式4）

【化5】



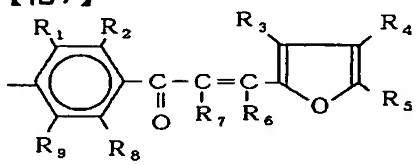
･･･（化学式5）

【化6】



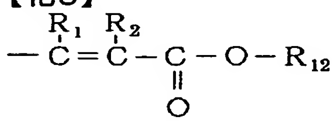
・・・(化学式6)

【化7】



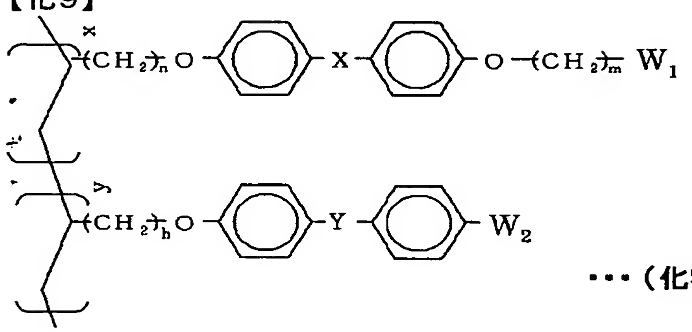
・・・(化学式7)

【化8】



・・・(化学式8)

【化9】



・・・(化学式9)

但し、$-R_1$～$-R_{11}$＝－H、ハロゲン基、－CN、アルキル基またはメトキシ基などのアルキルオキシ基、またはそれらを弗化した基、$-R_{12}$＝メチル基、エチル基などのアルキル基、またはそれらを弗化した基であり、x:y＝100～0:0～100、n＝1～12、m＝1～12、h＝1～12、X, Y＝none、－COO、－OCO－、－N=N－、－C=C－or－C_6H_4－、W_1, W_2＝化学式1または化学式2または化学式3または化学式4または化学式5または化学式6または化学式7または化学式8で表される構造である。

詳細な説明

【発明の詳細な説明】

【0001】

【発明の属する技術分野】感光性の重合体と低分子化合物の混合体の膜(フィルム)に非偏光性の紫外線を照射することによって、光軸を傾斜させて分子を配向させた位相差フィルムおよびその製造方法に関するものである。(特に、光軸がフィルム面に対し傾いた位相差フィルムは液晶表示装置において視野角拡大に有効である。)

【0002】

【従来の技術】位相差フィルムは、互いに垂直な主軸方向に振動する直線偏光成分を透過させ、この二成分間に必要な位相差を与える複屈折を有するフィルムである。このような位相差フィルムは液晶表示分野にも活用されてきており、特に光軸の傾いた位相差フィルムは光学補償フィルムとして液晶表示装置の視野角拡大に役立つ。このような位相差フィルムを製造する従来技術が幾つか報告されている。その一つとして、ポリカーボネートなどの高分子材料を延伸し、高分子鎖を配向させ、延伸方

向の屈折率と、延伸方向に対し直交方向の屈折率に差異を生じさせる方法であるが、分子が延伸方向に配向するため光軸を傾斜させることは実質的に不可能である。上記課題にかんがみ、光軸の傾いた位相差フィルムの製造法として延伸フィルムやラビング、光照射により配向処理した基材上で液晶性化合物を配列させる方法が提案または実用化されつつある。例えば、特開平7－287119号、特開平7－287120号公報では、ラビング配向膜、SiO斜方蒸着配向膜上にディスコティック液晶を配列させる方法が記載されている。また、同様な方法として、特開平10－278123号公報では光配向膜上に光重合開始剤を含有したディスコティック液晶を配向させ光照射によりこの配向を固定する方法が記載されている。上記のような配向膜を用いる方法では、配向膜の配向処理、液晶材料の配向など工程が煩雑になるなどの問題がある。更に、光軸の傾いた位相差フィルムを製造する他の方法として、無機誘電体を斜方蒸着する方法が提案されているが、長尺状シート上に連続して蒸着膜を形成するには、装置が大掛かりになったり、工程が煩雑になるなどの問題がある。光照射により位相差を発現させる方法として、特開平7－138308号にポリビニルシンナメートなどの感光性重合体に直線偏光性の(UV)光を照射する方法が記載されているが、該方法では照射した偏光UV光の電界振動と垂直方向に異方性が発現するため光軸を傾けることができないため、視野角を拡大し難い。また、本発明者も特開平10－278123号公報では感光性を有する側鎖型液晶性高分子への直線偏光性の紫外線照射により、光軸の傾いた位相差フィルムを製造する方法を提案した。しかし、光照射によって位相差を発現させる方法では、非偏光性の紫外線を直線偏光性に変換して照射する必要がある。このような偏光変換に用いられる一般的な2色性偏光子としては、PVA(ポリビニルアルコール)を一軸延伸したシートにヨードを含浸させたものをTAC(トリアセチルセルロース)で挟んだものがある。しかし、このようにヨードを含浸させた2色性偏光子では、紫外域の光の透過率や耐熱性が低いため液晶光配向技術としては使用には耐えない。このような理由から、紫外域の光を偏光させるには複屈折型プリズムが用いられているが、複屈折型プリズムでは方解石の自然結晶をプリズムとして用いるため、LCDに用いるような基板全面を照射できるような大型プリズムはない。

【0003】

【発明が解決しようとする課題】高分子フィルムの延伸配向によって作製された位相差フィルムでは、分子が延伸方向に配向するため光軸を傾斜させることが著しく困難である。一方、配向処理した基材上で液晶性化合物を配列させる方法や無機誘電体を斜方蒸着する方法は、光軸を傾斜させた位相差フィルムを作製することは可能であるが、工程が煩雑となるため低コストで大面積の光軸を傾斜させた位相差フィルムを得ることは容易ではない。また、直線偏光性の紫外線照射により位相差フィルムを製造する方法では偏光素子を介して照射光を直線偏光とする必要があるが、大面積を照射する場合の実用的な偏光素子を作製するのは困難である。

【0004】

【課題を解決する手段】本発明では、感光性の重合体と低分子化合物の混合体の膜(フィルム)に非偏光性の紫外線を照射することによって、光軸を傾斜させて位相差を発現させた位相差フィルムに関する。本発明の位相差フィルムの製造方法(による位相差フィルム)では、感光性の重合体と低分子化合物の混合体を製膜し、該膜面に対し特定の傾斜角をもって非偏光性の紫外線を照射することによって、膜中の分子を照射した該非偏光性の紫外線の傾斜角方向に傾けて配向させ、光軸を所望の方向に設定した位相差フィルムを提供できる。

【0005】

【発明の実施の形態】以下に、本発明の詳細を説明する。前述の感光性の重合体の例としては、液晶性高分子のメソゲン成分として多用されているビフェニル、ターフェニル、フェニルベンゾエート、アゾベンゼンなどの置換基と、シンナモイル基、カルコン基、シンナミリデン基、β－(2－フリル)アクリロイル基(または、それらの誘導体)などの感光性基を結合した構造を含む側鎖を有し、炭化水素、アクリレート、メタクリレート、マレイミド、N－フェニルマレイミド、シロキサンなどの構造を主鎖に有する高分子が挙げられる。該重合体は同一の繰り返し単位からなる単一重合体または構造の異なる側鎖を有する単位の共重合体でもよく、あるいは感光性基を含まない側鎖を有する単位を共重合させることも可能である。また、混合する低分子化合物も、メソゲン成分として多用されているビフェニル、ターフェニル、フェニルベンゾエート、アゾベンゼンなどの置換基を有する結晶性または、液晶性を有する化合物が挙げられる。混合する低分子化合物は、単一の化合物のみとは限らず複数種の化合物を混合することも可能である。更には、液晶性を損なわない程度に配向性を向上させるための配向助剤

や耐熱性を向上させるための架橋剤を添加することや、液晶性を損なうことなく液晶性を示さない単量体を感光性の重合体に共重合してもかまわない。但し、感光性の重合体と低分子化合物は、上記に限定されるものではない。

【0006】例を挙げて本発明を詳しく説明する。感光性の重合体と低分子化合物の混合体を塗布(スピンコートないしキャスト)し製膜する。該膜は、製膜時には等方性であり、感光性の重合体の側鎖部および低分子化合物は特定方向を向いていない。この状態を、図2に基づいて説明する。図2は、製膜後の膜を模式的に表したものである。膜(20)中では、長楕円で示される感光基を有する側鎖(2a、2b)および円柱で示される低分子化合物(2c)が無秩序に存在(共存)している。該膜に非偏光性の紫外線疑似平行光束(L)を照射すると、照射紫外線の進行方向と垂直方向に向いた側鎖(2a)は、平行方向を向いた側鎖(2b)より感光しやすいため、選択的に光反応が進む。これは、ベンゼン環などを含有する感光性部分の共役系が側鎖の長軸方向に延びているためであり、このような側鎖を光のような放射場に置いたとき、光の電界振動方向が側鎖の長軸方向と一致する場合に相互作用が極大となり、光の進行方向と側鎖の長軸方向が一致した場合には相互作用が極小となることによる。このことから、非偏光性の紫外線の照射により、特定方向の光反応を抑制した膜とすることができる。この光反応を進めるには、感光性基の部分が反応し得る波長の光の照射を要する。この波長は、感光性基の種類によっても異なるが、一般に200-500nmであり、中でも250-400nmの有効性が高い場合が多い。

【0007】図3は、図2の膜に非偏光の紫外線を照射した後の配向が促進された膜の模式図を示す。図3に示すように、膜(30)中の光反応を起こさなかった側鎖(3d)または低分子化合物(3c)は、非偏光性の紫外線の照射後の分子運動により、特定方向の光反応が抑制されたことにより膜中に発現した異方性の影響を受け配向する(側鎖(3a)は非偏光照射により感光し反応した側鎖、側鎖(3b)は照射紫外線の進行方向と平行方向を向いていたため反応しなかった側鎖をそれぞれ表している。)。その結果、膜全体において、非偏光性の紫外線の進行方向に傾けて配向させることができ、複屈折が誘起される。この照射を膜面に対して斜め方向から行なうと、光軸を任意に傾斜させて配向させることができるので、光軸を所望の方向に設定した位相差フィルムを提供できる。光軸の傾斜の測定には、Japan Journal Applied Physics, Vol. 19, 2013(1980)に掲載された、測定試料を回転させながら偏光の透過強度を測定するクリスタルローテーション法を用いた。該測定法では、偏光の透過率の角度依存性から測定試料の立体的な複屈折の測定ができる。非偏光性の紫外線の照射後の分子運動による配向は、膜を加熱すると促進される。膜の加熱温度は、光反応した部分の軟化点より低く、光反応しなかった側鎖と低分子の軟化点より高いことが望ましい。また、膜の配向を促進するには加熱下(室温からTi+5℃まで)で非偏光性の紫外線を照射することも有効である。ここで、Tiは液晶相から等方相へ変化するときの相転移温度を指す。好ましくはTi前後で非偏光性の紫外線を照射することが有効である。このように非偏光性の紫外線を照射したのち加熱、または加熱下で非偏光性の紫外線を照射したフィルムを該高分子の軟化点以下まで冷却すると分子が凍結され、本発明の位相差フィルムが得られる。

【0008】本発明において感光性の重合体に混合する低分子化合物が低分子化合物同士、もしくは該重合体に対して熱および/または光反応性を有している場合には、配向が強固に固定されるため耐熱性の向上が期待される。このような場合、再配向時の分子運動を妨げないよう、露光量を抑えるか反応性を調整するなどして、光反応点の密度を制御する必要がある。低分子化合物は、適量ならば曇り度を抑制する効果がある反面、過剰に添加すると曇り度の増加、配向性の低下を引き起こす。このような観点から、感光性の重合体または低分子化合物の種類にもよるが、低分子化合物を0.1wt%~80wt%添加しても位相差フィルムは製造可能であるが、好ましくは5wt%~50wt%であることが望ましい。ここで、重合体と低分子化合物の相溶性が十分でない場合には、製膜した時ないしは非偏光性の紫外線を照射した後の膜の加熱によって相分離や可視光の散乱を誘起しうる大きさの結晶を生成し曇り度の増加の原因となる。

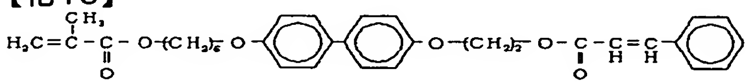
【0009】膜厚を厚くしより大きな位相差のフィルムを得る手法として、膜を積層する方法が挙げられる。この場合には、先に製膜して非偏光性の紫外線を照射した膜上に材料溶液を塗布し積層する工程で、この先に形成された膜の破壊を防ぐために、溶解性を下げた溶媒に重合体および低分子化合物を溶解し用いることが有効である。また、感光性化合物の膜に表裏面から非偏光性の紫外線を照射すると、複屈折がより効率よく発現するようになる。感光性の化合物は支持体上に塗布するなどし

て製膜され、非偏光性の紫外線の照射は化合物に直接または支持体を介してもよい。支持体を介する場合には、支持体は感光性の化合物の反応しうる波長の光の透過性を有している限りどのような材料でも良いが、光透過率が高い程、照射量が少なくて済み、製造工程上有利となる。また、剥離性の支持体上で感光性の化合物を製膜し、剥離後、膜の表裏面より非偏光性の紫外線を照射することもできる。

【0010】感光性重合体の原料化合物および低分子化合物に関する合成方法を以下に示す。

（単量体1）4，4'ービフェニルジオールと2ークロロエタノールを、アルカリ条件下で加熱することにより、4ーヒドロキシー4'ーヒドロキシエトキシビフェニルを合成した。この生成物に、アルカリ条件下で1，6ージブロモヘキサンを反応させ、4ー（6ーブロモヘキシルオキシ）ー4'ーヒドロキシエトキシビフェニルを合成した。次いで、リチウムメタクリレートを反応させ、4ーヒドロキシエトキシー4'ー（6ーメタクリロイルヘキシルオキシ）ビフェニルを合成した。最後に、塩基性の条件下において、塩化シンナモイルを加え、化学式10に示される単量体1を合成した。

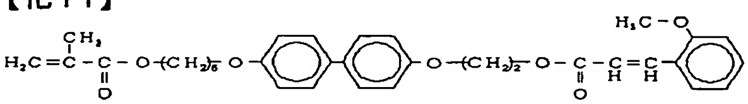
【化10】



・・・（化学式10）

【0011】（単量体2）4，4'ービフェニルジオールと2ークロロエタノールを、アルカリ条件下で加熱することにより、4ーヒドロキシー4'ーヒドロキシエトキシビフェニルを合成した。この生成物に、アルカリ条件下で1，6ージブロモヘキサンを反応させ、4ー（6ーブロモヘキシルオキシ）ー4'ーヒドロキシエトキシビフェニルを合成した。次いで、リチウムメタクリレートを反応させ、4ーヒドロキシエトキシー4'ー（6ーメタクリロイルヘキシルオキシ）ビフェニルを合成した。最後に、塩基性の条件下において、2ーメトキシ塩化シンナモイルを加え、化学式11に示される単量体2を合成した。

【化11】



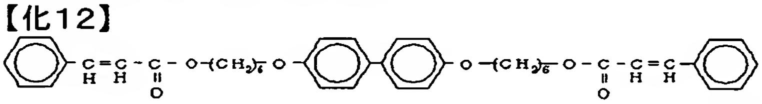
・・・（化学式11）

【0012】（重合体1）この単量体1をテトラヒドロフラン中に溶解し、反応開始剤としてAIBN（アゾビスイソブチロニトリル）を添加して重合することにより感光性の重合体1を得た。この重合体1は、47ー75℃の温度領域において、液晶性を呈した。

【0013】（重合体2）この単量体2をテトラヒドロフラン中に溶解し、反応開始剤としてAIBNを添加して重合することにより感光性の重合体2を得た。この重合体2も液晶性を呈した。

【0014】（低分子化合物1）4，4'ービフェニルジオールと6ーブロモヘキサノールを、アルカリ条件下で反応させ、4，4'ー ビス（6ーブロモヘキシルオキシ）ビフェニルを合成した。次いで、塩基性の条件下において、塩化シンナモイルを加え反応させ、生成物をカラム精製することにより化学式12に示される低分子化合物1を合成した。

【化12】



・・・（化学式12）

【0015】図1には、本発明の位相差フィルムの製造方法（装置）を、例を挙げて示す。電源（12）によって励起された紫外線ランプ（11）で発生した非偏光性の紫外線（16）を、支持体（15）上に塗布（コート）された感光性の重合体と低分子化合物の混合体の膜（14）に照射する。実施例1から3は、本発明の製造法により光軸の傾いた位相差フィルムを作製した実施例である。

【0016】（実施例1）3．75重量％の重合体1および1．25重量％の液晶材料E7（メルクジャパン）をジクロロエタンに溶解し、石英基板（支持体）上に約3μmの厚さで塗布した。該基板を水平面に対して45度傾け、塗布面が照射面となるように配置し、非偏光性の紫外線を、水平面に対し垂直方向から

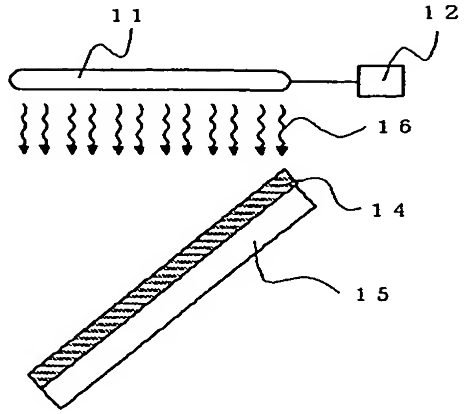
室温で120mJ／cm^{2}照射し、続いて、基板を裏返し同様に非偏光性の紫外線を120mJ／cm^{2}照射した。次に、100℃に加熱した後、室温まで冷却した。このようにして得られた基板は、光軸が基板の法線方向から20°傾き、基板面内の位相差は14nmであった。
【0017】（実施例2）3．75重量％の重合体2および1．25重量％の液晶材料E7（メルクジャパン）をジクロロエタンに溶解し、石英基板（支持体）上に約3μmの厚さで塗布した。該基板を水平面に対して45度傾け、塗布面が照射面となるように配置し、非偏光性の紫外線を、水平面に対し垂直方向から室温で120mJ／cm^{2}照射し、続いて、基板を裏返し同様に非偏光性の紫外線を120mJ／cm^{2}照射した。次に、100℃に加熱した後、室温まで冷却した。このようにして得られた基板は、光軸が基板の法線方向から25°傾き、基板面内の位相差は20nmであった。
【0018】（実施例3）3．75重量％の重合体1および1．25重量％の低分子化合物1をジクロロエタンに溶解し、石英基板（支持体）上に約3μmの厚さで塗布した。該基板を水平面に対して45度傾け、塗布面が照射面となるように配置し、非偏光性の紫外線を、水平面に対し垂直方向から室温で120mJ／cm^{2}照射し、続いて、基板を裏返し同様に非偏光性の紫外線を120mJ／cm^{2}照射した。次に、100℃に加熱した後、室温まで冷却した。このようにして得られた基板は、光軸が基板の法線方向から7°傾き、基板面内の位相差は1nmであった。
【0019】これら実施例により作製された光軸の傾いた位相差フィルムに、更に紫外線を照射することにより未反応の感光性基の光反応を促進させ、フィルム中の配向を強固に固定することができる。このような位相差フィルムは、耐熱性、光安定性に優れ実用に充分であった。これらの実施例から、非偏光性の紫外線を照射することにより光軸を制御したフィルムを作製でき、従来技術のように実用的な偏光素子を用いなくとも液晶表示装置の視野角拡大に有効な光軸方向を制御した位相差フィルムの製造が可能であることが立証された。
【0020】
【発明の効果】非偏光性の紫外線の照射という簡便な操作により、従来技術のような延伸工程を用いなくても位相差フィルムを得ることができる。更に、非偏光性の紫外線の照射方向を変えることにより同一基板内において、光軸の異なる領域の作製も可能であり、様々な光学素子への活用が期待される。また、光軸の傾斜した位相差フィルムは、旋光モード、複屈折モードを利用したねじれネマチック液晶を使った液晶表示装置において視野角拡大用の光学補償フィルムとして活用できる。従来このような、光軸の傾斜した大面積の位相差フィルムを低コストで作製できなかったが、本発明によって、斜め方向から非偏光性の紫外線を照射するという簡便な操作で大面積化が可能となった。
【0021】

図の説明

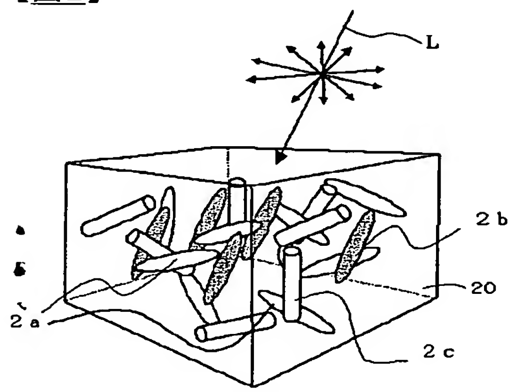
【図面の簡単な説明】
【図1】本発明の位相差フィルムの製造方法を示す概念図
【図2】非偏光性の紫外線の照射により感光した側鎖を示す模式図
【図3】非偏光性の紫外線を照射した後の分子運動により配向したフィルムを示す模式図
【符号の説明】
11・・・紫外線ランプ
12・・・電源
14・・・膜（フィルム）
15・・・基材（支持体）
16・・・紫外線（非偏光性の光）

図面

【図1】



【図2】



【図3】